CDS olutions

APPLICATIONS INFORMATION USING ADVANCED SAMPLE HANDLING TECHNOLOGY

Pyrolysis-Catalytic Hydrogenation of Vegetable Oil

Vegetable oils generally contain triglycerides of C_{16} and C_{18} fatty acids. The fatty acids are relatively volatile, so when they are freed by pyrolysis they mostly appear intact, with some fragmentation, as shown in Figure 1. A pulse pyrolysis of vegetable oil performed in hydrogen looks much the same, since the pyrolysis is more rapid than the hydrogenation.

To pyrolyze a sample and completely hydrogenate the pyrolysis products requires a two-step approach involving a catalyst and reactor. In the first step, the original sample is pyrolyzed, generating volatiles that are carried in hydrogen to the reactor. The second step takes place in the reactor which contains a reducing catalyst and is independently temperature controlled. A sorbent trap is used between the pyrolysis-hydrogenation zone of the instrument and the GC to reduce pressure and switch from the reactant gas (hydrogen) to GC carrier (helium). This permits experiments using the reactor pressure as high as 500 PSI, collecting the products onto the trap and then thermally desorbing them to the GC.

Figure 2 shows a sample of the same vegetable oil, again heated to 700°C for pyrolysis. The pyrolysis products are then carried to a 300°C reactor containing a platinum catalyst, operated at 30 PSI. Here the pyrolysate is thoroughly hydrogenated producing only saturated hydrocarbons.

Figure 1 shows that the main products from just pyrolysis are palmitic and oleic acids, plus many unsaturated hydrocarbons. When the experiment includes the hydrogenation reactor, however, the product distribution is very different. Figure 2 shows that the main products now are a distribution of normal hydrocarbons, containing 11 to 18 carbons, with octadecane being the largest single peak.

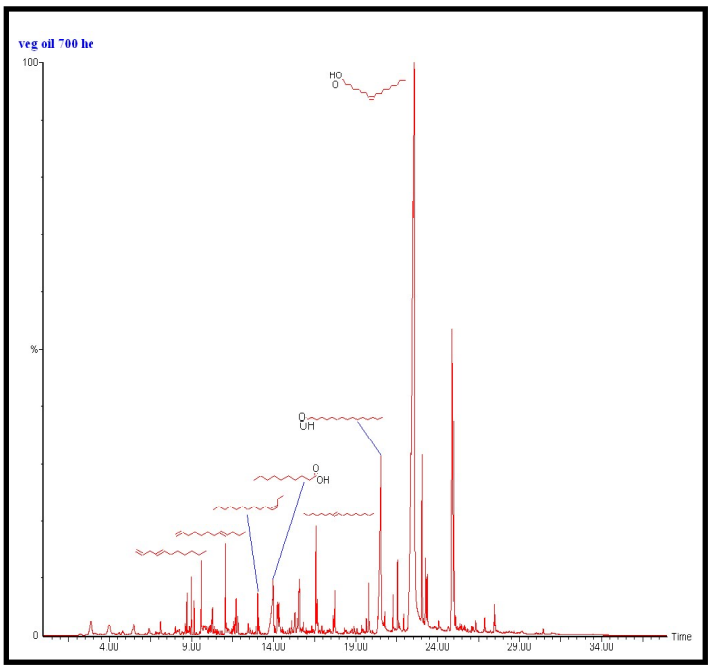

Figure 1. Py-GC/MS of vegetable oil in helium.

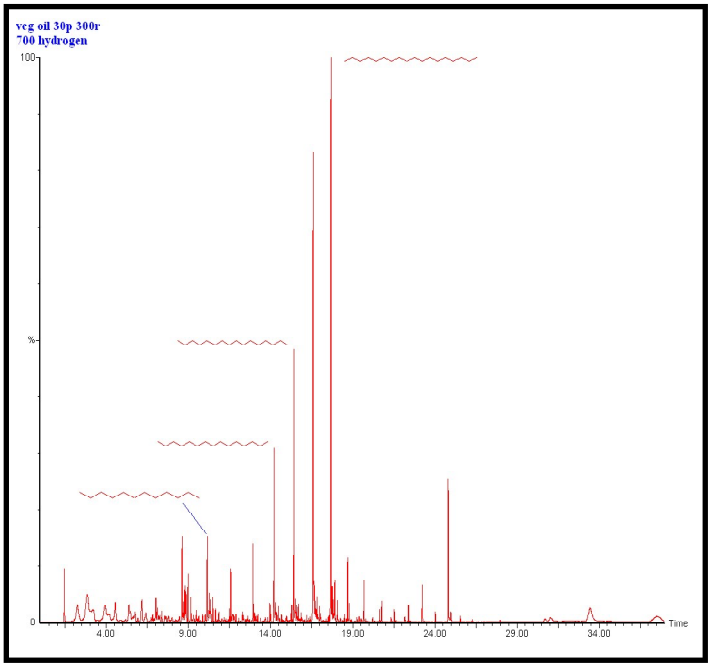

Figure 2. Py-hydrogenation of vegetable oil.

Experimental Conditions

Samples were applied as a film to a quartz rod and pyrolyzed in the coil of a CDS Pyroprobe 5200.

Pyrolysis

Temperature:	700°C for 15 seconds
Interface:	300°C for 4 minutes
Valve oven:	300°C
Transfer line:	300°C
Trap desorption:	300°C for 4 minutes

Hydrogenation

Reactor temp.:	300°C
Carrier:	Hydrogen
Pressure:	30 PSI

Chromatography

Column:	30 m x 0.25 mm 5% phenyl methyl silicone
Oven:	40°C for 2 minutes 10°C/minute to 300°C
Carrier:	Helium
Split:	50:1
Mass range:	35 to 550 amu

FOR MORE INFORMATION CONCERNING THIS APPLICATION, WE RECOMMEND THE FOLLOWING READING:

S. Tsuge et al., Structural characterization of polyolefins by pyrolysis-hydrogenation glass capillary gas chromatography, J. Anal. Appl. Pyrolysis, 1, 1980, 221 - 229.

Additional literature on this and related applications may be obtained by contacting your local CDS Analytical representative, or directly from CDS at the address below.

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